

# PATENT SPECIFICATION

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## COMPLETE SPECIFICATION

### Improvements in or relating to Rubber-base Adhesive

We, ARMSTRONG CORK COMPANY, a Corporation organised under the Laws of the State of Pennsylvania, United States of America, of Lancaster, State of Pennsylvania, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

10 This invention relates to adhesives, and more particularly to rubber-base adhesives. Still more particularly it relates to a rubber-base adhesive possessing excellent heat fatigue resistance and a good tack range.

15 The preparation of rubber-base adhesives containing polychloroprene (neoprene) admixed with other substances is well known; solutions of neoprene and butadiene-styrene blends are known. Although such prior adhesives have been useful in many applications, their usefulness has been limited to those uses requiring excellent heat fatigue resistance and good tack range.

It is an object of the present invention to 25 supply such an adhesive.

According to the present invention, an adhesive is prepared by blending together a first constituent comprising a rubbery linear polymer capable of curing in the final stages 30 to a cross linked polymer, such as chloroprene, and a second constituent capable of curing by the chemical addition of sulfur, such as a butadiene-styrene copolymer, for example, GR-S. The blended mass is then heated in 35 the absence of sulfur to a temperature sufficient to cause cross linking of the first constituent. The resultant product is then dispersed in a solvent.

The first constituent comprising a rubbery 40 linear polymer capable of curing in the final stages to a cross-linked polymer is preferably chloroprene. Other suitable rubbers meeting this definition are the polyacrylic rubbers. The second constituent capable of curing by 45 the chemical addition of sulfur is preferably

one of the butadiene-styrene rubbers such as GR-S. Other examples of the second constituent are the Buna N's.

Based on 100 parts by weight of the blend of the two rubbers, the maximum amount of 50 the first constituent should be about 75 parts by weight. Greater amounts of the first constituent produce a mass that may be dispersed during mixing but which quickly separates upon standing. Lesser amounts 55 may be used, however, without causing any difficulty. Preferably there should be at least about 5 parts by weight of the first constituent per 100 parts by weight of the total rubber blend in order that the final ad- 60 hesive may possess the highly desirable increased heat fatigue resistance. Thus the rubber blend may have a composition which varies from about 75 parts of the first constituent with about 25 parts of the second 65 constituent to about 5 parts of the first constituent with about 95 parts of the second constituent.

The mixing of the two types of rubber is preferably carried out in a Banbury mixer. 70 In order that the mass may be prevented from breaking up under the mixing action and, further, to impart tack and adhesion, the mixing and curing is preferably carried out in the presence of a resin. Suitable resins may 75 be generally described as those having compatibility with both rubbers and melting or softening points under 325°F. Examples of such resins are stabilized rosin, terpene-phenolic resins, cumar resins, and terpene 80 resins.

By proper selection of resins to be blended with the two rubbery constituents, adhesives of different physical properties can be prepared, varying from those that are perman- 85 ently tacky to those with extraordinarily high heat resistance and substantially shorter tack range. The amount of resin or resin mixture to be added to the rubber blend can vary from about 5 parts by weight resin per 100 90

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parts rubber blend to about 150 parts by weight resin per 100 parts rubber blend. The preferred range is 50-110 parts by weight resin per 100 parts by weight rubber blend.

- 5 The resins and rubbery constituents are hot mixed in an internal type mixer to a temperature in the range of about 230°-325°F. Mixing is carried out in the absence of sulfur until the cure of the first constituent is substantially complete. The time of mixing will depend on the temperature used within the range outlined above. A longer time will be required for temperatures only slightly above the minimum of 230°F., at which temperature the cure of the first constituent such as chloroprene takes place at a slow rate. The temperature should not exceed the upper limit of about 325°F., since to do so causes decomposition of the chloroprene, oxidation of the GR-S, and loss of volatile compounding agents.

- Since the composition of the present invention contains no sulfur and since the curing temperature is controlled, it can be seen that the present process is one in which one of two rubbers in the blend can be cured under conditions that permit the other to remain basically unchanged. At elevated temperatures, the chloroprene, for example, undergoes curing by cross-linking, while the butadiene-styrene copolymer remains substantially unchanged. By bringing about this selective curing, the rubber mass will increase substantially in cohesive strength, stiffness, and elasticity but will still retain the ability of the uncured mass to be wet by and dispersed in those solvents normally used to dissolve butadiene-styrene rubbers. By varying the proportions of the two constituents as outlined above, it is possible to vary the cohesive strength and elasticity of the final adhesive. Amounts of chloroprene near the maximum amount as described above yield an adhesive of increased cohesive strength and decreased elasticity. Amounts of chloroprene near the minimum described above yield an adhesive of decreased cohesive strength and increased elasticity.

- There may also be added during the blending of the two constituents accelerators such as zinc oxide, litharge, and the common organic accelerators. Fillers may be utilized, as for example Loomite talc, ZnO, clay, whiting, asbestos, and carbon black.

- 55 The final mass is preferably dispersed in a rubber solvent. Examples of such solvents are Shell rubber solvent, Gulf VM&P naphtha, toluol, and xylol. These solvents are preferably added after cure of the first constituent has taken place and when the blended mass has been cooled somewhat. The amount of solvent to be added may vary between wide limits, but it is preferred that the amount of solvent be such that the solids content of the final adhesive should be in the

range of about 20-70% by weight.

The following examples illustrate the invention. All parts listed are by weight unless otherwise noted.

#### EXAMPLE I.

Into a Banbury type mixer there is placed:

GR-S 1013	.. .. .	35.0 parts	
Neoprene W	.. .. .	65.0	"
3 NO (accelerator)	.. .. .	37.0	"
Accelerator 833	.. .. .	0.5	"
Agerite Stalite (antioxidant)	.. .. .	1.0	"
" Polypale " Resin (Stabilized v. rosin)	.. .. .	62.0	"

The word "Polypale" is a registered Trade Mark.

The above mixture was blended for 30 minutes at a temperature of 300°F. At the end of that period of time, the mix was cooled and there was added 245 parts naphtha as a rubber solvent. When dispersion was complete, the product proved to be an adhesive having extraordinary heat fatigue resistance and a tack range of about 2 hours.

#### EXAMPLE II.

Into a Banbury there is placed:

GR-S 1009	.. .. .	35.0 parts	
GR-S 1013	.. .. .	35.0	"
Neoprene W	.. .. .	9.5	"
ZnO	.. .. .	3.5	"
Accelerator 833	.. .. .	0.3	"
DBPC (antioxidant)	.. .. .	1.5	"
Loomite talc	.. .. .	13.0	"
Isoester "A" (stabilized rosin)	.. .. .	6.0	"

and the mixture was blended for 35 minutes at 300°F. On cooling, there was added 65 more parts Isoester "A" plus 30 parts Herculyn (tackifier). On dissolving the mass in 250 parts rubber solvent, the resultant adhesive was permanently tacky.

What we claim is:—

1. A method of making a rubber-base adhesive which comprises blending together a first constituent comprising a rubbery linear polymer capable of curing in the final stages to a cross-linked polymer and a second constituent capable of curing by the chemical addition of sulfur, the first constituent being present in an amount of about 5-75 parts by weight and the second constituent being present in an amount of about 95-25 parts by weight, heating said blend in the absence of sulfur to a temperature sufficient to cause cross-linking of said first constituent, and dispersing the resultant product in a solvent.
2. A method according to Claim 1, wherein said first constituent comprises a chloroprene and said second constituent comprises a butadiene-styrene rubber.
3. A method according to Claim 1 or 2, wherein said temperature is in the range of about 230°-325°F.
4. A method according to any one of Claims 1, 2 or 3, wherein a tackifier resin compatible with said first and second constituents is blended with said first and second

constituents.

5. A method according to Claim 4, wherein a filler and an accelerator are blended with the said constituents and tackifier resin.

5 6. A method according to Claim 4 or 5, wherein the first constituent is present in about 5-75 parts by weight, the second constituent in about 95-25 parts by weight, and the tackifier resin in an amount in the range  
10 of about 5-150 parts by weight resin per 100 parts by weight of the mixture of said constituents.

7. A method according to any one of the

preceding claims, wherein the resultant product is dispersed in the solvent to a solids content of about 20-70% by weight.

8. A method of making a rubber-base adhesive substantially as hereinbefore described.

9. A rubber-base adhesive whenever made 20 by the method of any one of the preceding claims.

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